

BEST AVAILABLE COPY

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau(43) International Publication Date
31 October 2002 (31.10.2002)

PCT

(10) International Publication Number
WO 02/085237 A2

(51) International Patent Classification⁷: A61C

(21) International Application Number: PCT/US02/14050

(22) International Filing Date: 25 April 2002 (25.04.2002)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
01042829 25 April 2001 (25.04.2001) UA
not yet assigned 9 November 2001 (09.11.2001) CA

(71) Applicant (for all designated States except US): GENERAL PLASMA, LLC [US/US]; 240 Elkins Lane, P.O. Box 436, Lusby, MD 20657 (US).

(72) Inventors; and

(75) Inventors/Applicants (for US only): KIRYUKHIN, Mykola, M. [UA/UA]; 16 Fesenkovskaya St., ap.9, Kharkov, 61068 (UA). ZABASHTA, Lidiya, O. [UA/UA]; 39 Zhukova St., ap.6, Kharkov, 61022 (UA). BABENKO, Victor, O. [UA/UA]; 15 Krala, ST., ap.2, Kharkov 61075

(UA). VOLKOV, Yuyiy, Y. [UA/UA]; 3 Mira Boulevard, ap.32, Kharkov, 61108 (UA). STRELNYTSKYI, Volodymyr, Y. [UA/UA]; 4 Eliszarova St., ap.281, Kharkov, 61098 (UA).

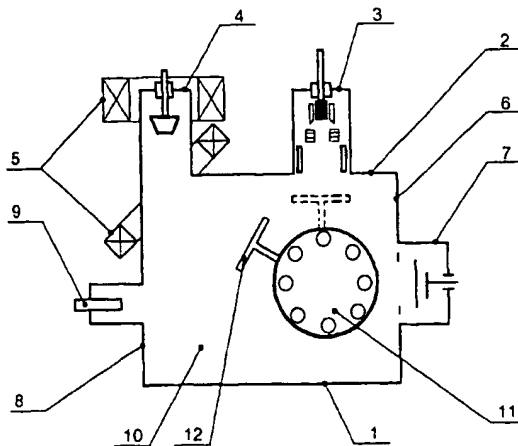
(74) Agents: STONE, Gregory, M. et al.; Whiteford, Taylor & Preston L.L.P., Seven Saint Paul Street, Baltimore, MD 21202-1626 (US).

(81) Designated States (national): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW.

(84) Designated States (regional): ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

[Continued on next page]

(54) Title: DIAMOND-LIKE COATING, METHOD OF ITS PLATING AND DENTAL BUR WITH THE SAID DIAMOND-LIKE COATING



(57) Abstract: A layer of diamond-like substance is plated on a product surface by way of carbon deposition in the gas phase under the conditions of reduced pressure, the layer comprising an aggregate of successive sub-layers of diamond-like coating with reduced concentration of contaminants in the interface areas of the sub-layers. The layers are plated in a sealed chamber under pressures lower than atmospheric pressure and under controlled temperature. The method entails first plating a metal sub-layer from a separate stationary source of metal plasma, followed by plating the diamond-like coating on products that are held under floating voltage. Prior to powering each of the metal and carbon plasma sources, the respective plasma flow is completely blocked with a shutter, the plasma source is turned on, and after a predetermined amount of time, the shutter is opened. The coating is particularly suitable for use on dental burs.

WO 02/085237 A2

WO 02/085237 A2**Published:**

— *without international search report and to be republished upon receipt of that report*

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

WO 02/085237

PCT/US02/14050

**DIAMOND-LIKE COATING, METHOD OF ITS PLATING
AND DENTAL BUR WITH THE SAID DIAMOND-LIKE COATING**

5

Technical Field

The invention is related to the field of producing highly rigid wear-resistant coatings on metal and non-metal goods, in particular on medical cutting tools, for example, on dental burs.

10

Background Art

Diamond-like coatings (DLC) are widely used in various branches of science and technology. Some of the main advantages of DLC's are their hardness, wear-resistance, purity, biological inertness, etc. Due to such properties DLC's are plated on medical tools, medical implants, and cutting tools, and are likewise used in other
15 branches.

There is known a DLC plated by vacuum arc method disclosed in the USSR Certificate of Authorship No. 1070949 comprising a DLC layer on a substrate, the DLC being plated by a pulse stream of compensated no-current carbon plasma, the pulse stream having a density of 10^{18} - 10^{19} cm⁻²c⁻¹ (particles per square centimeter per
20 second).

However, the disclosed coating does not have the necessary thickness due to poor DLC adhesion to the substrate.

The closest prior art for the coating according to the invention claimed is a coating disclosed in the USSR Certificate of Authorship No. 1494554 published on
25 March 15, 1994, Bulletin No. 5. The coating has an increased strength of the sub-layer adhesion to the substrate and to the following layer of DLC. Yet strength provided by

WO 02/085237

PCT/US02/14050

this method is not sufficient for producing a DLC with high wear resistance due to a high concentration of contaminants in the DLC.

There is known a method for producing highly rigid DLC's on metal and dielectric substrates by way of cathode spraying of graphite in a magnetic field at low pressure of the inert gas krypton (10^{-5} - 10^{-2} Pa) on the substrate being cooled and having the temperature lower than 100 K (the USSR Certificate of Authorship No. 411037 published in 1974, Bulletin No. 29). A drawback of this method is poor quality of the coating, low productiveness, and high cost of the products.

Main efforts of researchers and manufacturers of DLC's are aimed at increasing their wear resistance defined both by adhesion to the substrate and the structure of the coating itself. One of the most important parameters of the DLC deposition process is the substrate (product) temperature that is not to exceed the critical temperature of 135-150 °C during the process, because at higher temperatures, coatings start losing their physical and mechanical properties and adhesion to the substrate due to thermal graphitization. Furthermore, it is necessary to obtain high purity of the source material, high density of the carbon flow, high purity of the substrate surface, etc. As a result, a pulse ion-plasma vacuum arc (further – vacuum arc) method of plating DLC was developed, the essence of which lies in forming an intensive pulse flow of carbon ions in an electric arc in a vacuum.

One of the known vacuum arc methods of DLC plating is a method disclosed in the USSR Certificate of Authorship No. 1070949 that involves DLC condensation on the substrate produced by a pulse flow of compensated no-current carbon plasma with density of 10^{18} - 10^{19} cm⁻²c⁻¹.

However, the said method does not provide for the necessary thickness of the coating due to poor DLC adhesion to the substrate.

WO 02/085237

PCT/US02/14050

The closest prior art for the method according to the claimed invention is a method disclosed also in the USSR Certificate of Authorship No. 1494554 published on March 15, 1994, Bulletin No. 5, the essence of which lies in the fact that before vacuum arc DLC plating on the substrate surface, the said surface is coated by a sub-layer of metal, e.g. titanium having increased strength of adhesion to the substrate and the following layer of DLC, the product being under floating voltage during the process of plating the sub-layer of metal and DLC.

However, adhesion strength obtained by this method is still insufficient for producing a DLC with high wear resistance. Besides, it is reasonable to plate the titanium sub-layer only on metal substrates whereas DLC may be plated on non-metal substrates as well.

One of the applications of strengthening coatings is their plating on medical cutting tools, in particular on dental burs. Known are dental burs made of steel or hard-alloyed materials (for example, see the State Standard of Ukraine – 22090-89). Yet these burs have a short durability due to insufficient hardness and wear resistance of cutting edges.

The closest prior art for dental burs claimed according to this invention is dental burs of Romidan Dental Ltd. presented on the Internet at the address: <http://www.romidan.com/fgburs.htm>. The heads of these burs, which are made of stainless steel, are coated by electrostatic gluing with a coating of diamond powder made of natural diamonds. Drawbacks of these burs are their high cost and relatively short durability due to the fact that strength of the bond between the powder and metal is determined by the glue adhesion to the powder and metal.

WO 02/085237

PCT/US02/14050

Disclosure of Invention

As far as the DLC and method of its plating are concerned, this invention is based on the objectives of improving wear resistance of the product due to the increase in the level of the coating adhesion to the substrate and increasing the coating
5 thickness due to the change of its structure.

As far as dental burs are concerned, this invention is based on the objective of increasing their wear resistance by introducing new materials and elements.

The set objective as to the coating is solved as follows: the disclosed coating comprises a diamond-like substance layer plated on the substrate by carbon deposition
10 in the gas phase under reduced pressure conditions, said layer being an aggregate of successive sub-layers of diamond-like coating with the reduced concentration of contaminants in the areas of the sub-layers interface.

The decrease in the concentration of contaminants is obtained as follows: during the process of plating the coating on the product surface by deposition of a
15 carbon plasma flow from a pulse arc source in a sealed chamber at pressures below atmosphere pressure, before turning on the carbon plasma source, the plasma flow is blocked with a shutter, then the source is turned on, and then in the predetermined period of time the shutter is opened. Then a relatively thin DLC sub-layer is plated, the source is turned off, the shutter is closed, and the process is repeated.

20 The improvement claimed in this invention consists in the creation of the coating structure out of a number of successive sub-layers with a sharp decrease in concentration of contaminants both at the interface of the successive sub-layers and within one sub-layer. This increases adhesion of sub-layers to each other and to the substrate and so allows to increase the DLC quality and thickness that in turn
25 increases the coating strength and wear resistance.

WO 02/085237

PCT/US02/14050

A further improvement of the coating according to the invention is an additional titanium sub-layer plated by titanium deposition in the gas phase before plating the diamond-like coating on the steel substrate. Adhesion of the whole coating to the substrate is increased due to the fact that titanium has increased adhesion to metal, especially to steel, as a metal. A particular effect is obtained when before plating titanium the titanium plasma flow is blocked with a shutter for a certain period of time. This also leads to decrease in amount of contaminants at the interface and to increase in the DLC adhesion to the substrate and wear resistance of the coating.

In specific embodiments of the invention as a result of the research performed, the following parameters of the coating were optimized:

thickness of the diamond-like coating sub-layer is not less than 0.1 μm ;

amount of the diamond-like coating sub-layers is within the range of 1 – 20.

The objective set as to the method is solved by adding new operations to the known method of DLC plating on the product surface comprising plating a diamond-like coating on the product surface by deposition of a carbon plasma flow from a pulse arc source, the said process taking place in a sealed chamber under pressures lower than atmospheric pressure, the temperature of the product being controlled during the deposition process, and the product being under floating voltage, said new operations being as follows: before turning on the carbon plasma source this source is completely blocked with a shutter, then the source is turned on, and in the predetermined period of time the shutter is opened.

The blocking of the carbon plasma source with a shutter before turning it on does not allow the carbon plasma flow to be deposited on the products, and it is these initial periods when a great amount of contaminants, which impregnate the cathode during free air entering into the chamber and during the process of pumping out by oil

WO 02/085237

PCT/US02/14050

pumps, are penetrating into the plasma when leaving the surface of the carbon plasma source cathode made of graphite and being a porous material with a well-developed surface. Contaminants present in initial layers of coatings reduce significantly the adhesion of one layer to another one and impair the coating quality. Therefore, the improvement claimed in this invention reduces sharply the quantity of contaminants getting on the product surface and thereby increases the metal adhesion to the product surface and to the DLC, which in turn increases wear resistance of the DLC.

A further improvement of the method according to the invention is that before plating a diamond-like coating on metal products, a sub-layer of metal is plated on the product surface from a separate stationary source of metal plasma, the said source being blocked with a shutter before turning it on, then this source is turned on, and in the predetermined period of time the shutter is opened.

As it is known, a sub-layer of metal, e.g. titanium, has an enhanced adhesion to the substrate metal, and on the other hand, a sub-layer of metal plated by the aforesaid method has an increased purity and therefore has an enhanced adhesion to the following sub-layer of DLC.

Besides, similar to the case with the carbon plasma source, the shutter blocking the metal plasma flow after turning on the source does not allow this metal plasma flow to be deposited on the substrate during initial periods of the source operation when the amount of contaminants in the flow is the highest.

Thus, this improvement further increases the DLC adhesion to the substrate.

In a further improvement of the method according to the invention, before plating a metal sub-layer on the product surface, a separate stationary ion source of argon is turned on, and ion bombardment of products with argon ions is performed during the predetermined amount of time. Such a bombardment heats up at least the

WO 02/085237

PCT/US02/14050

surface layer of the products and contributes to cleaning the surface from contaminants entering the external space. This further increases the metal adhesion to the product surface.

In a specific embodiment of the bombardment of products with argon ions, the energy of argon ions is established within the range of 5 – 6 keV, and a negative potential is applied to the products within the range of 800 – 1,500 V.

In a specific embodiment of the method, which comprises plating a metal sub-layer, after turning on the metal plasma source with the closed shutter the arc current is maintained within the range of 90 – 100 A, and the duration of the predetermined period of time, after which the shutter of the metal plasma source is opened, is established within the range of 50 – 60 seconds. These ranges were determined experimentally to be the most appropriate ones for the maximum removal of contaminants from the metal plasma source cathode.

A further improvement of the method according to the invention, which comprises plating a metal sub-layer, is that during the said predetermined period of time a separate stationary ion source of argon is turned on, and ion bombardment of products with argon ions is performed with the energy 5 – 6 keV, while a potential is applied to products within the range of 1,400 – 1,500 V. The effect of the bombardment on the degree of the metal sub-layer adhesion to the product surface is described above, and performing the bombardment during the metal plasma source preparation for operation reduces the period of plating the coating. The aforesaid ranges of physical values are also determined experimentally and are the most appropriate ones for the said operation.

In a specific embodiment of the method, which comprises plating a metal sub-layer, this sub-layer is plated with the negative potential on the products being within

WO 02/085237

PCT/US02/14050

the range of 180 – 200 V. The said range is optimum for plating a metal sub-layer and is also determined experimentally.

A further improvement of the method according to the invention is to repeat the process of the diamond-like coating plating several times with plating a sub-layer of the coating each time, wherein each time before turning on the source of carbon plasma the plasma flow is blocked completed with a shutter, then the source is turned on, and in the predetermined period of time the shutter is made open. This allows to obtain reduced concentration of contaminants in every sub-layer interface area, which decreases stresses in this area and enables to increase the coating strength and wear resistance to a further extent.

In a specific variant of any of the aforesaid embodiments of the method according to the invention, the duration of the predetermined period of time, after which the shutter is opened, is within the range of 40 – 80 seconds. This experimentally determined period of time is sufficient for contaminants to be removed to the utmost from the surface of the carbon plasma source cathode.

By experimental research of conditions of DLC plating on products without exceeding the critical temperatures, specific values of the process parameters were chosen, namely:

- energy of carbon ions not exceeding 0.4 keV;
 - duration of the carbon source pulses being 10 μ sec with the pulse period-to-pulse duration ratio not less than 30,000;
- which may be used in the method according to the invention both separately and in the aggregate.

A further improvement of the method according to the invention is that before opening the pulse carbon source with a shutter it is turned off, and the ion source of

WO 02/085237

PCT/US02/14050

argon is turned on for the predetermined period of time. This allows cleaning of the product surface before plating the next sub-layer on products due to the increase in their temperature, and enhances the adhesion of the following DLC layer to the preceding one.

- 5 In specific embodiments of the method, wherein a number of sub-layers of the coating are plated, the thickness of each sub-layer of the diamond-like coating is not less than 0.1 μm and/or the amount of sub-layers is within the range of 1 – 20.

- A further improvement of the method according to the invention is to introduce an additional new operation of argon ion bombardment of the product
10 surface from a separate ion source during the process of the DLC deposition from a pulse source of carbon plasma.

- In the known methods of producing DLC with the help of a pulse source of carbon plasma it was impossible to produce a coating more than 1.5 μm thick due to poor adhesion and large internal stresses in the coating, while many practical
15 applications require coatings up to 5 μm thick and more.

 As it is known, the DLC's produced by the known methods have a columnar structure having large internal stress when the coating is sufficiently thick, which results in the cracking of coating at the border of grains and in its detachment from the substrate with the great thickness of the coating.

- 20 Furthermore, one of the causes explaining the quality impairment of the DLC plated by the said method on small-sized products with sharp cutting edges and poor thermal conductivity is the local heating of products to temperatures higher than critical temperature, which results in graphitization of metastable diamond structure of the coating and disastrous reduction of wear resistance and strength of the coating.
25 During the bombardment of the product surface with argon ions obtained from a

WO 02/085237

PCT/US02/14050

separate ion source, which takes place simultaneously with the DLC deposition from the pulse carbon source, the argon ions irritate the columnar structure, which contributes to more uniform stress distribution in the coating and reduces the internal stress appearing in the coating during the deposition, whereby graphitization of the structure is avoided. Thus increased quality of the coating is provided, and it is possible to increase its thickness.

By experimental research of conditions of DLC plating on products without exceeding the critical temperatures the energy of the doping argon gas ions was chosen to be within the range of 4 – 6 keV in the specific embodiment of the invention.

The objective set in this invention as to the dental bur improvement is solved as follows: in a dental bur comprising a wear-resistant coating layer the coating is a diamond-like coating comprising a number of successive sub-layers with reduced concentration of contaminants in the interface areas of the sub-layers.

The technical result obtained thereby lies in the increase in wear resistance of burs due to the plating continuous and homogeneous coating and to the increase in the coating adhesion to the bur material.

The improvements and specific embodiments of the dental bur according to the invention are similar to those described above for the coating.

20

Brief Description Of The Drawings

Other objects, features, and advantages of the present invention will become more apparent from the following detailed description of the preferred embodiment and certain modifications thereof when taken together with the accompanying drawings in which:

25

WO 02/085237

PCT/US02/14050

Figure 1 is a top view of an apparatus for applying the coating according to the instant invention.

Best Mode(s) for Carrying Out the Invention

5 The subjects of the invention are described below by way of examples of operation of the unit, the horizontal section of which is schematically illustrated in the attached drawing, and which is a vacuum chamber with an evacuation system for high vacuum (not shown). The front wall 1 is a sealed door through which products are loaded into the chamber, and on the back wall 2 there are: a pulse source of carbon
10 plasma 3 and a stationary source of metal plasma 4 with a magnetic deflection system 5. On the right wall 6 of the chamber there is an argon ion source 7, and on the left wall 8 there is an infrared pyrometer 9 for the product temperature control. On the upper (not shown) and bottom wall 10 of the chamber there are two identical rotators 11. The shutter 12 is made rotatable around the axis of the rotators 11 and able to
15 block flows both of metal and carbon plasma (the position is shown with a dotted line) as well as to be placed in the neutral position in which no flow and hairline of the pyrometer are blocked.

Example 1. Plating coatings on cutters.

Pre-polished steel cutters are rinsed in an alkaline solution in an ultrasound
20 bath and then in distilled water by way of immersion. Then they are rinsed in benzene in an ultrasound bath and wiped dry with unbleached cotton napkins. Further they are fixed on holders of rotators in the vacuum chamber, then the chamber is closed, and evacuation starts. Upon obtaining pressure in the chamber not exceeding $2 \cdot 10^{-3}$ Pa, a rotator drive is turned on, and ion bombardment of products with argon ions with the
25 energy of 5 – 6 keV is performed for 10 – 12 minutes, the rotator being under

WO 02/085237

PCT/US02/14050

negative potential of 800 – 1500 V. After argon irritation the titanium cathode surface of the metal plasma source is being cleaned. For that an arc is initiated in the source with the current of 90 – 100 A, which burns during 60 seconds with the closed shutter. Following that the potential of 1500 V is applied to the product, and the shutter is

5 opened without turning off the arc current. At that the products are irritated with the metal plasma during 50 – 60 seconds under the condition of continuous product temperature control with the help of the infrared pyrometer, which is followed by the potential reduction to 180 V, and a titanium sub-layer is plated on the product during 3 minutes. After that the titanium source is turned off, and the potential is cut off from

10 the products, the products becoming insulated together with the rotator (under the floating voltage). Following that the products are left for 7 – 10 minutes to cool to the temperature less than 120 °C. Then the graphite cathode of the pulse carbon source is being cleaned, for which purpose it is turned on with the closed shutter and degassed for 60 – 80 seconds, the pulse propagation frequency being equal to 3 – 10 Hz. The

15 graphite cathode readiness for operation is indicated by restoration of vacuum in the chamber not worse than $2 \cdot 10^{-3}$ Pa. After that the carbon source is turned off, the argon ion source is turned on, and the product is irritated for 3 minutes for deletion of contaminants that appeared on it during the cathode degassing, following which the pulse carbon source is turned on without turning off the argon ion source, the shutter

20 is opened, and the DLC is deposited on the products. During the DLC deposition the following parameters are maintained: pulse duration – 10 μ sec, reservoir capacitor voltage – 400 – 450 V, pulse propagation frequency – 1 – 2 Hz, argon ion source voltage – 4 kV with the current of 50 A, pressure in the chamber - $(2.1 - 2.4) \cdot 10^{-2}$ Pa, continuous product temperature – 80 – 100 °C. With these parameters the DLC

25 deposition speed is 4 μ m per hour. The coating was deposited for 2 hours taking into

WO 02/085237

PCT/US02/14050

account the fact that the process was interrupted for the products to cool to the temperature less than 100 °C, the coatings being formed without scabbing and delaminating. Wear resistance of cutters with the coatings deposited under the said conditions increased by 3 – 5 times.

5 Example 2. Plating the DLC on dental burs.

Pre-polished steel burs are rinsed first in benzene in an ultrasound bath and then with rectified alcohol by way of immersion, following which they are wiped dry with cambric or unbleached cotton napkins. Further they are fixed on holders of rotators in the vacuum chamber, the chamber is closed, and evacuation starts. Upon
10 obtaining pressure in the chamber not exceeding $2 \cdot 10^{-3}$ Pa a rotator drive is turned on, and ion bombardment of products with argon ions with the energy of 5 – 6 keV is performed from the argon ion source for 8 minutes. At that potential of 0 – 1500 V is gradually (0, 800, 1500 V) applied to the rotator. After argon irritating the metal (titanium) cathode surface of the metal plasma source is being cleaned. For that at
15 first the shutter is placed in the position blocking the metal plasma source, and an arc is initiated therein with the current of 100 - 120 A, which burns during 50 seconds. After that, potential of 1500 V is applied to the products, the shutter is placed in the position blocking the carbon plasma source 3, and the metal plasma irritating of the products is performed for 20 – 60 seconds at the continuous product temperature
20 control with the help of the infrared pyrometer, which is followed by the potential reduction to 180 - 200 V, and a metal (titanium) sub-layer is plated on the products during 3 minutes. Then the titanium source is turned off, and the potential is cut off from the products, the products becoming insulated together with the rotator (under the floating voltage). After that the graphite cathode of the pulse carbon source is
25 cleaned by the above-mentioned method, for which purpose it is turned on with the

WO 02/085237

PCT/US02/14050

closed shutter and degassed for 40 – 80 seconds, the pulse frequency being equal to 3 – 10 Hz. The graphite cathode readiness for operation is indicated by restoration of vacuum in the chamber not worse than $2 \cdot 10^{-3}$ Pa. Further, the carbon source is turned off, the argon ion source is turned on, and the products are irradiated for 30 – 60
5 seconds for deletion of contaminants that appeared on them during the cathode degassing, following which the pulse carbon source is turned on without turning off the argon ion source, the shutter is opened and placed in neutral position, and the DLC is deposited on the products. Upon obtaining the coating sub-layer thickness of 0.1 μm the sources are turned off for products to cool to the predetermined
10 temperature, and the operations are repeated. The coating was deposited for 2 hours until the thickness of 2 μm was obtained, and it had 20 sub-layers and was formed without apparent scabbing and delaminating. Wear resistance of burs under these conditions increased in 3 – 15 times depending on the quality of the original product.

Thus, a DLC and method of its plating are provided, the DLC having
15 increased adhesion to the product, reduced concentration of contaminants at the interfaces of the sub-layers, owing to which internal stress in the DLC is reduced, a possibility to increase the DLC thickness is provided, and its wear resistance is enhanced.

The thickness of one sub-layer of the coating is determined by the quantity of
20 contaminants in the original products, their usual quantity enabling to grow a sub-layer not less than 0.1 μm thick.

The amount of sub-layers is determined by the coating purpose, and, as it was discovered experimentally, the range of the layer amount from 1 to 20 is sufficient for the majority of applications.

25 One application of the invented coating is its plating on dental burs.

WO 02/085237

PCT/US02/14050

The invented coating can be plated on any metal and non-metal products, in particular, on any cutting tools in order to increase their wear resistance.

Industrial Applicability

5 For the industrial application of coatings for metal and non-metal products, it is desirable to provide coatings which are highly rigid and wear resistant. Herein disclosed is a wear resistant diamond-like coating for metal and non-metal products comprising layers of a diamond-like substance plated on the product to be coated by way of carbon deposition in the gas phase under the conditions of reduced pressure to
10 produce an aggregate of successive sub-layers with reduced concentration of contaminants in the sub-layer interface areas. The layers are plated in a sealed chamber under pressures lower than atmospheric pressure and under controlled temperatures by the method of plating a metal sub-layer from a separate stationary source of metal plasma, and further plating the diamond-like coating on the products
15 under floating voltage. Prior to turning on each of the metal and carbon plasma sources, the plasma flow is completely blocked with a shutter, after which the plasma source is turned on, and after a predetermined amount of time, the shutter is opened.

WO 02/085237

PCT/US02/14050

CLAIMS

1. A diamond-like coating comprising a layer of diamond-like substance
plated on a substrate by way of carbon deposition in the gas phase under the
5 conditions of reduced pressure, said layer further comprising an aggregate of
successive sub-layers of diamond-like coating.
2. The diamond-like coating of claim 1, further comprising a titanium sub-
layer plated by way of titanium deposition in the gas phase before the diamond-like
10 coating plating.
3. The diamond-like coating of claim 1, wherein the thickness of each sub-
layer of diamond-like coating is not less than 0.1 μm .
- 15 4. The diamond-like coating of claim 1, wherein the amount of sub-layers of
diamond-like coating is within the range of 1 to 20.
5. A method for plating a diamond-like coating on a product surface
comprising the steps of:
20 placing a first shutter between a stationary metal plasma source and a product
surface;
turning on said stationary metal plasma source;
after a first predetermined amount of time, moving said first shutter out of a
direct path extending from said stationary metal plasma source to said product
25 surface;

WO 02/085237

PCT/US02/14050

plating a metal sub-layer on a product surface from a stationary metal plasma source;

placing a second shutter between a carbon plasma source and a product surface;

5 turning on said carbon plasma source;

after a second predetermined amount of time, moving said second shutter out of a direct path extending from said carbon plasma source to said product surface; and

plating a diamond-like coating on said product surface by way of deposition of a carbon plasma flow from a pulse arc source;

10 wherein said plating and coating steps are performed in a vacuum chamber having pressures lower than atmospheric pressure while controlling the product temperature, and maintaining the product under floating voltage.

6. The method of claim 5, further comprising the step of repeating said step of
15 plating a diamond-like coating on said product surface several times by successively plating a sub-layer of the coating, blocking the carbon plasma flow with a shutter before turning on the carbon plasma flow source, turning on said carbon plasma flow source, and opening said shutter after said second predetermined period of time.

20 7. The method of claim 6, wherein the thickness of each sub-layer of diamond-like coating is not less than 0.1 μm .

8. The method of claim 6, wherein the amount of sub-layers of diamond-like coating is within the range of 1 to 20.

25

WO 02/085237

PCT/US02/14050

9. The method of claim 5, wherein said first and second predetermined period of time is between 40 and 80 seconds.

10. The method of claim 5, further comprising the step of bombarding the product surface with argon ions from a separate stationary ion source simultaneously with the step of plating a diamond-like coating on said product surface.

11. The method of claim 10, wherein the energy of said argon ions is maintained within the range of 4 – 6 keV during said step of bombarding the product surface with argon ions.

12. The method of claim 5, wherein the energy of carbon ions during said step of plating a diamond-like coating on said product surface does not exceed 0.4 keV.

13. The method of claim 5, wherein the duration of carbon source pulses during said step of plating a diamond-like coating on said product surface does not exceed 10 μ sec, and the pulse period-to-pulse duration ratio is not less than 30,000.

14. A dental bur comprising a layer of wear-resistant coating, said layer of wear resistant coating further comprising a diamond-like coating comprising an aggregate of successive sub-layers.

15. The dental bur of claim 14, further comprising a titanium sub-layer plated by way of titanium deposition in gas phase underneath of said diamond-like coating.

25

WO 02/085237

PCT/US02/14050

16. The dental bur of claim 14, wherein each sub-layer of said diamond-like coating is not less than 0.1 μm .

17. The dental bur of claim 14, wherein the amount of sub-layers of diamond-
5 like coating is within the range of 1 to 20.

18. A method of plating a diamond-like coating on a product surface comprising the steps of:

placing a first shutter between a carbon plasma pulse arc source and a product
10 surface;

turning on said carbon plasma pulse arc source;

after a first predetermined amount of time, moving said first shutter out of a direct path extending from said carbon plasma pulse arc source to said product surface; and

15 plating a diamond-like coating on said product surface by way of deposition of a carbon plasma flow from said pulse arc source;

wherein said plating and coating steps are performed in a vacuum chamber having pressures lower than atmospheric pressure while controlling the product temperature, and maintaining the product under floating voltage.

20

19. The method of claim 18, further comprising the steps of:

prior to said step of placing a first shutter between said pulse arc source and said product surface, placing a second shutter between a stationary source of metal plasma and a product surface, turning on said stationary metal plasma source, after a

25 second predetermined amount of time moving said second shutter out of a direct path

WO 02/085237

PCT/US02/14050

extending from said stationary metal plasma source to said product surface, and
plating a metal sub-layer on said product surface from said stationary metal plasma
source.

5 20. The method of claim 19, further comprising the step of turning on a
separate stationary ion source of argon and bombarding said product surface with
argon ions during said second predetermined amount of time.

21. The method of claim 20, wherein the energy of said argon ions is within
10 the range of 5-6 keV, and a negative potential is applied to the products within the
range of 800-1,500V while the product surface is bombarded with argon ions.

22. The method of claim 19, further comprising the step of:
after turning on said stationary metal plasma source and while said second
15 shutter is positioned between said stationary metal plasma source and said product
surface, maintaining an arc current within the range of 90-100 A, and said second
predetermined amount of time is within the range of 50-60 seconds.

23. The method of claim 19, further comprising the step of turning on a
20 separate stationary ion source of argon and bombarding said product surface with
argon ions during said second predetermined amount of time, while maintaining the
energy of the argon ions within the range of 5-6 keV and applying a potential to the
products within the range of 1,400 – 1,500 V.

WO 02/085237

PCT/US02/14050

24. The method of claim 19, further comprising the step of maintaining a negative potential on the products within the range of 180 – 200 V while plating said metal sub-layer on said product surface.

5 25. The method of claim 18, further comprising the step of repeating said step of plating a diamond-like coating on said product surface several times by successively plating a sub-layer of the coating, blocking the carbon plasma flow with a shutter before turning on the carbon plasma flow course, turning on said carbon plasma flow source, and opening said shutter after said first predetermined period of
10 time.

26. The method of claim 25, wherein the thickness of each sub-layer of diamond-like coating does not exceed 0.1 μm .

15 27. The method of claim 25, wherein the amount of sub-layers of diamond-like coating does not exceed 20.

28. The method of claim 18, wherein said first predetermined amount of time is within the range of 40-80 seconds.

20

29. The method of claim 18, wherein the energy of carbon ions during said step of coating said product surface with a diamond-like coating does not exceed 0.4 keV.

WO 02/085237

PCT/US02/14050

30. The method of claim 18, wherein the duration of carbon source pulses during said step of coating said product surface with a diamond-like coating does not exceed 10 μ sec, and the pulse period-to-pulse duration ratio is not less than 30,000.

5 31. The method of claim 18, further comprising the step of:
before moving said first shutter, turning off said carbon plasma pulse arc source, and turning on a separate stationary ion source of argon for said first predetermined amount of time.

10 32. The method of claim 18, further comprising the step of bombarding the product surface with argon ions simultaneously with the step of plating a diamond-like coating from a pulse source of carbon plasma on said product surface.

15 33. The method of claim 32, wherein the energy of argon ions is maintained within the range of 4-6 keV.

WO 02/085237

PCT/US02/14050

1/1

FIGURE 1

